

DERWENT-ACC-NO: 1993-405562

DERWENT-WEEK: 199350

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TITLE: Coating for machine tools - comprises nickel@, aluminium@, vanadium@, oxygen and nitrogen, and is obtd. by three electrode cathodic sputter process

on static jig with biasing electrode to remove impurities

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PRIORITY-DATA: 1992US-0893261 (June 4, 1992)

PATENT-FAMILY:

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MAIN-IPC			
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IT LU MC N

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APPLICATION-DATA:

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APPL-DATE		
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INT-CL (IPC): B32B009/00; B32B019/00 ; C23C014/34

ABSTRACTED-PUB-NO: WO 9324316A

BASIC-ABSTRACT: The coating on a surface-coated article consists of at least

Ti, Al, V, O and N. Also claimed is a process of coating a substrate by placing

it proximate to an anode in a housing also including a cathodic target mfd.

from at least Ti, Al and V, evacuating the housing to 0.1 to 0.001 torr and

then introducing O2 and N2 while imposing a potential between target and anode

to form the coating.

The coating also includes Mo and Y and is $2.0-8.0 \times 10$ power minus 5 and esp.

6.5×10 power minus 5 thick. Ti, Al and V, opt. Mo, are present in a nitride

or oxide form. The target comprises Ti, Al, Mo and V and the potential between

it and the anode is 500-1000 V. A bias electrode is included and has a potential relative to the cathode of 22.0-23.0 below the potential between

anode and cathode. The vacuum is maintained at 0.01 torr and the gases are

introduced in the wt. ratio 1-1.25 pt. O2 to 1 pt. N2. The cathode comprises

concentric rings with the substrate located between them.

USE/ADVANTAGE - The substrate is a machine tool mfd. from steel, stainless steel, titanium, carbide or inconel alloys and is uniformly coated over its surface, or the substrate is Al₂O₃, Si₃N₄ or SiO₂. Other typical substrates are airfoils and turbine blades. In a three electrode cathodic sputtering process, a durable Ti-contg. coating is obtd. using a stationary jig and such that the presence of a biasing electrode reduces inclusion of impurities in the coating.

CHOSEN-DRAWING: Dwg.1/1

DEFWENT-CLASS: L02 M13 P73 X25

EPI-CODES: L02-F02; L02-F03; L02-J01C; M13-G01;

EPI-CODES: X25-A04;

----- KWIC -----

ABTX:

The coating on a surface-coated article consists of at least Ti, Al, V, C and

N. Also claimed is a process of coating a substrate by placing it proximate to

an anode in a housing also including a cathodic target mfd. from at least Ti,

Al and V, evacuating the housing to 0.1 to 0.001 torr and then introducing O₂

and N₂ while imposing a potential between target and anode to form the coating.

ABTX:

The coating also includes Mo and Y and is 2.0-8.0 x 10 power minus 5 and esp.

6.5 x 10 power minus 5 thick. Ti, Al and V, opt. Mo, are present in a nitride

or oxide form. The target comprises Ti, Al, Mo and V and the potential between

it and the anode is 500-1000 V. A bias electrode is included and has a potential relative to the cathode of 22.0-23.0 below the potential between

anode and cathode. The vacuum is maintained at 0.01 torr and the gases are

introduced in the wt. ratio 1-1.25 pt. O₂ to 1 pt. N₂. The cathode comprises

concentric rings with the substrate located between them.

ABTX:

USE/ADVANTAGE - The substrate is a machine tool mfd. from steel, stainless

steel, titanium, carbide or inconel alloys and is uniformly coated over its

surface, or the substrate is Al₂O₃, Si₃N₄ or SiO₂. Other typical substrates

are airfoils and turbine blades. In a three electrode cathodic

sputtering
process, a durable Ti-contg. coating is obtd. using a stationary jig
and such
that the presence of a biasing electrode reduces inclusion of
impurities in the
coating.

TTX:

COATING MACHINE TOOL COMPRISE NICKEL@ ALUMINIUM@ VANADIUM@ OXYGEN
NITROGEN
OBTAIN THREE ELECTRODE CATHODE SPUTTER PROCESS STATIC JIG BIAS
ELECTRODE REMOVE
IMPURE

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TITLE: OCR SCANNED DOCUMENT
DATE-ISSUED: May 8, 1973

US-CL-CURRENT: 204/298.05, 204/298.06 , 427/523 , 427/569

May 8, 1973 J. S. PRZYBYSZEWSKI ET AL 39732,158 METHOD AND APPARATUS
FOR
SPUTTERING UTILIZING AN APERTURED ELECTRODE AND A PULSED SUBSTRATE BIAS
Filed
Jan. 14 1971 0 MATCHIN 18 OFK RADIO FREQUENC POWER SOURCE 32 16 12 28
34 T 30
10 2 1 HIGH OLTAGE D.C. (0-5 KV.) 24 1 4 INVENTORS JOHN S.
PRZYBYSZEWSKI
RICHARD K. SHALTENS BY ATTORNEYS
United States Patent Office 3973211 59 3,732,158 METHOD AND APPARATUS
FOR
SPUTTERING UTILIZING AN APERTURED ELECTRODE AND A PULSED SUBSTRATE BIAS
John S.
Przybyszewski, North Olmsted, and Richard K. 5 Shaltens, Lakewood,
Ohio,
assignors to the United States of America as represented by the
Administrator
of the National Aeronautics and Space Administration Filed Jan. 14,
1971, Ser.
No. 106,424 Int. Cl. C23c 15100 10 U.S. Cl. 204-192 7 Claims ABSTRACT
OF THE
DISCLOSURE Combinin-g the advantages of ion plating with the
ver-satility of a
radio frequency sputtered source. A pulsed high 1,5 voltage direct
current is
passed to the article being plated during radio frequency sputtering.
ORIGIN
OF THE INVENTION 20 The invention described herein was made by
employees of the
United States Government and may be manufactured and used by or for
the
Government for governmental purposes without the payment of any
royalties
thereon or 25 therefor. BACKGROUND OF THE INVENTION This invention is
concerned
with plating adherent films particularly directed to ion plating
alloy films
on such objects using a radio frequency sputtered source. The ion p
lating
process is modified because this source supplies film material at a much
slower
rate than the usual thermal evaporation source. 3 5 In the past
several
methods were used to deposit various types of films on simple as well
as
geometrically complex objects. While each process is satisfactory for

certain applications, problems have been encountered with all of these methods. 40

Conventional vapor deposition is conducted in a vacuum of 10^{-6} to 10^{-8} torr. The use of conventional vapor deposition has been generally restricted to the elemental metals, although some metal alloy systems as well as certain semiconductors and nonconductors have been vapor deposited. The high vacuum used in vapor deposition reduces the concentration of gas molecules which increases the mean free path. Very little scattering of the film material results, and the process is limited to line-of-sight deposition. The coating of complex geometries by vapor deposition is conditioned on the rotation of the object to be coated. Adherence of a vapor deposited film is poor because of the low energy of the impinging film material. The film adhesion is improved when either direct current or radio frequency sputtering is used. Direct current sputtering has been successful for depositing elemental metals, semiconductors, and metal alloy systems. This type of sputtering is not useful for depositing nonconductors. Radio frequency sputtering has been used for depositing elemental metals, metal alloy systems, semiconductors, and nonconductors. This type of sputtering is not limited by the nature of the film material. It can be used to sputter almost any material from insulators through semiconductors to metals. 6,5

Radio frequency and direct current sputtering are generally done in an atmosphere having a pressure in the range of about 5 to 20 microns. Because of this relatively high pressure the sputtered material is scattered. The mean free path is short, and the material is diffused rapidly as it leaves the source. Even though this scattering effect causes film formation on surfaces not directly facing the source, a tenfold may be 8, 1973 2 material, both RF and DC sputtering are considered to be line-of-sight deposition processes. The low energy of the impinging film material adversely affects film adherence. Ion plating is performed at about the same pressure as RF and DC sputtering. A high voltage is applied to the object to be coated. This results in a uniform coating on all sides without rotating or moving

either the object or the source of film material. When the coating has excellent adhesion, problems have been encountered because, the process utilizes a thermal evaporation source. This limits the film materials to the elemental metals and those compounds which do not dissociate before they evaporate. SUMMARY OF THE INVENTION These problems have been solved by the present invention which utilizes radio frequency sputtering with a pulsed high voltage direct current. The process is not limited to a line-of-sight deposition, and complex geometries can be plated without rotation. The process is useful for plating adherent films of elemental metals, metal alloy systems, semiconductors, and nonconductors. OBJECTS OF THE INVENTION It is, therefore, an object of the present invention to plate an adherent alloy film on an object having a geometrically complex configuration. Another object of the invention is to provide an improved plating method which combines the advantages of thermal source. A further object of the invention is to provide an improved method for plating alloy films on complex geometries without rotation during the plating process. These and other objects of the invention will be apparent from the specification which follows and from the drawing wherein like numerals are used throughout to identify like parts. DESCRIPTION OF THE DRAWING The figure is a schematic diagram of a system constructed in accordance with the invention for plating adherent alloy films on geometrically complex objects. DESCRIPTION OF THE PREFERRED EMBODIMENT Referring now to the drawing there is shown an object 10 which is to be coated in accordance with the present invention. The object 10 may be any electrically conductive article having either a simple or geometrically complex configuration. By way of example the invention has been utilized to coat bearings with a solid lubricant. The object 10 is mounted in a chamber 12 that is connected at 14 to a suitable vacuum pumping system. A target 16 of the material to be sputtered is likewise located in the chamber 12. The target 16 is connected to a radio frequency power source 18 through a matching

network 20. This RF sputtered source 16 is utilized instead of a thermal evaporation source normally used in ion plating. As stated earlier, certain modifications are required because the RF sputtered source 16 supplies film material at a much slower rate than a thermal evaporation source. A suitable gas is supplied to the chamber 12 at an inlet 22. Argon is preferably used. The object 10 to be plated is connected to a high voltage, direct current source 24. The source 24 preferably has a range of 0 to 5 kilovolts. In this manner the ion plating is carried out in a low pressure ionized gaseous atmosphere with the object 10 to be plated forming a cathode that is maintained at a high negative potential from the source 24. Consequently, the object 10 to be plated is continuously bombarded or sputtered by ions before, during and after film material on objects having complex geometries. The invention is an ion plating with the versatility of a radio frequency sputtering process enters the ionized gas. If the evaporation rate of the film material is too slow the film on the object to be plated will be sputtered away as fast as it develops, and no film will result. Because the RF sputtered source 16 is inherently slow, no film would develop under normal ion plating conditions. According to the present invention the ion plating process has been modified to reduce the rate of sputtering off of the newly formed film by providing a timed switch 26 between the source 24 and the object 10. The reduction in the sputtering off rate is accomplished by pulsing the negative high voltage DC from the source 24 to the object 10 by means of the timed switch 26. A third electrode 28 is positioned in the chamber 12 to establish a common electrode between the RF power source 18 and the DC power source 24. The electrode 28 is connected to the high voltage DC power source 24 through an RF choke 30. The electrode 28 forms an anode with respect to the cathode 10. The radio frequency power source 18 is connected to the electrode 28 through a capacitor 32. A bypass capacitor 34 is likewise provided. This third electrode 28 is preferably in the form of a perforated plate or screen that is located between the sputtered source 16 and the object 10. The screen has an aperture

in the center to enable sputtered material to pass to the object to be plated. In operation, the object 10 is mounted in the chamber 12 together with the target 16 of material to be sputtered. The chamber 12 is partially evacuated and a gaseous atmosphere from about 10-20 microns pressure is established. A high voltage DC negative potential of 2 to 5 kilovolts with respect to the screen 28 is continuously applied to the object 10. This establishes a glow discharge within the vacuum chamber 12 to sputter clean the object 10. After a predetermined period of sputter cleaning, the high voltage DC source 24 is deenergized and the gaseous pressure is lowered to about 1,0 microns. The RF power source 18 to the film material 16 is energized, and sputtering of the film material begins. At this point the high voltage DC source 24 is switched to a timed on-off mode by the switch 26. In this manner the high voltage DC is reenergized and reapplied to the object 10. The pulsed high voltage direct current RF sputtering process produces an intense electric field which completely surrounds the object 10. This can be seen as a dark space around this object. Any ionized material entering this region gains kinetic energy from the field and impacts on the surface of the object 10 with great force. This contributes to improved adhesion. The dark space, in effect, represents essentially a source of film material takes on the general outline of the object 10. The high voltage DC negative potential on the object 10 is maintained throughout the plating. The process is continued until the desired film is obtained. The reduced sputtering rate of the film on the object 10 results in the formation of a visible film having excellent adherence and covering the entire object. EY,AMPLE Pulsed hi_eh voltage direct current radio frequency sputtering was used to plate antifriction bearing components with a solid lubricant film of molybdenum disulfide. The plating conditions are as follows: Total coating time each component: 3 hours Radio Frequency Input Power to Source Material: 700 watts at 7 megahertz Maximum pulse amplitude to specimen- 2000 V-DC, negative Pulse form: 15 seconds on; 5 minutes off; 5: duty cycle Specimen to screen distance: 2.5 inches 317321158 4 Specimen to source

distance: approximately 6 inches Chamber pressure: 5 microns; argon The components were assembled and the bearing was tested. The bearing was satisfactory for its intended use. .5 While one embodiment of the invention has been shown and described it will be appreciated that various modifications to the invention may be made without departing from the spirit of the invention or the scope of the subjoined claims. 10 What is claimed is: 1. In a sputtering apparatus, including a vacuum chamber, means for admitting a gas into said chamber, target holding means for supporting the material to be sputtered, substrate holding means for supporting a substrate 15 to be coated, means for applying RF potential to said target to sputter said material, and means for applying a high voltage direct current to said substrate; the improvement wherein an apertured electrode is disposed between said substrate holding means and said 20 target holding means, said electrode being connected to said means for applying RF potential and said means for applying a high voltage direct current whereby a high voltage direct current negative potential with respect to said electrode is applied to 2.5 said substrate holding means and an RF potential is applied to said target holding means to sputter said material, and timed switch means connected to said means for applying a high voltage direct current, said timed switch 30 means enabling said high voltage direct current negative potential to be pulsed from of about 15 seconds on and about 5 minutes off. 2. Apparatus as claimed in claim 1 wherein said apertured electrode is a screen. 35 3. Apparatus as claimed in claim 2 wherein the screen has a centrally disposed aperture therein. 4. In an RF sputtering process wherein an RF potential is applied to a target to sputter material from the target onto a substrate, the improvement comprising disposing 40 an apertured electrode between the target and the substrate, connecting said electrode to a source of RF potential and to a source of high voltage direct current, and applying to said substrate a high voltage direct current negative active potential pulse from of about 15 seconds on and 45 9 about 5 minutes off. 5. A method as claimed in claim 4 wherein said pulsed voltage is a potential of about 2 to 5 kilovolts. 6. A method as claimed in claim 4 including disposing

50 said target about 2.5 inches from said apertured electrode and said substrate about 6 inches from said apertured electrode. 7. A method as claimed in claim 6 wherein argon is utilized at a pressure of about 10 microns.

55 References Cited UNITED STATES PATENTS 3,361,659 1/1968 Bertelsen

----- 204-298 3,526,584 9/1970 Shaw ----- 204-298 60
3,528,906

9/1970 Cash et al ----- 204-295 3,461,054 8/1969 Uratny

204-192 3,479,269 11/1969 Byrnes et al - ----- 204-192 3,530,055
9/1970

Maissel et al ----- 204-192 3,589,994 6/1971 Schwartz et al -

204-192 65 JCHN H. MACK, Primary Examiner S. S. KANTER, Assistant
Examiner

U.S. Cl. X.F. 70 204-298